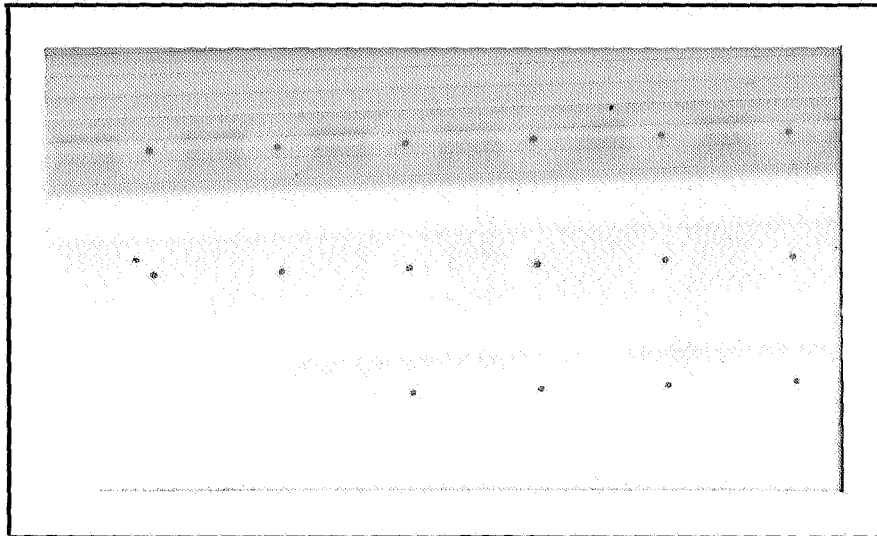


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REPORT NO. 13  
SEMI-ANNUAL PROGRESS REPORT  
1 July 1968 to 31 December 1968  
STUDIES IN FUNDAMENTAL CHEMISTRY  
OF FUEL CELL REACTIONS  
NsG-325

Submitted to:  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
Washington, D.C. 20546

Submitted by:  
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## SECTION I

Title of Project: Electrochemical Instrumentation and Experimental Techniques

Long-term Aims: The development of new techniques and the refinement of old techniques in experimental electrochemistry.

Specific Aims for this Period:

(1) The electronic (digital) portions of the precision sound velocity meter have been completed, and function according to expectations. The limiting factor at present is the machined screw for acoustic pickup translation, and a new lapped lead-screw is being fabricated.

(2) The gated oscillator for ultrasonic EMF measurements has been completed and is operating properly. Some preliminary measurements of the dependence of the U. E. M. F. show that some artifactual difficulties may still remain.

(3) It has been discovered that tungsten bronzes can be melted under the right conditions, and that it should be possible to grow single crystals from the melt by standard metallographic growing techniques. Crystals larger than 1 mm have already been produced, but more careful control will be required for larger crystals. Work is under way to

this end.

(4) Triangular-wave sweep studies of the electrochemical behavior of the tungsten bronzes indicate an anomalous capacitance near the  $H_2$  evolution potential, probably due to an absorption pseudo-capacitance, and possibly indicating proof of the existence of a hydrogen form of the bronzes.

(5) Attention has been directed to the acquisition of an on-line/time-sharing computer facility for the group. The proposed system utilizes a teletype-writer as the remote terminal. Besides general utility for the group (remote programming in a conversational mode), the system can be used for the direct processing of Mossbauer data, since the unit which we have outputs data on a compatible teletype system.

(6) A new type of cell has been developed and built for the metal-metal oxide studies which lowers by more than an order of magnitude the residual IR drop for potentiostatic and/or galvanostatic measurements. Residual IR's of less than  $0.05 \Omega$  corresponding to a working-reference spacing of less than  $40 \mu$ . Additional electronics is required to utilize these low residual IR drops and is being developed.

Other:                   The course in "Electronics and Instrumentation for  
Electrochemistry" has been started. Approximately  
10 lectures have been given to date.



## SECTION II

Title of Project:	Theoretical Study of Electrocatalysis
Long-term Techno-logical Aims:	To establish quantum mechanical expressions for the cases which would apply to electrocatalytic ethylene oxidation and oxygen reduction.
Specific Aims for this Period:	Critical review of the theoretical works on the homogeneous and heterogeneous redox reactions in aqueous solution. Preliminary review of the works on the structural characteristics of the electrode-solution interface.
Specific Aims for the Next Report Period:	To investigate how to incorporate the characteristics of the double layer structure in the electron transfer expressions. To explore electronic interaction between the electrode surface and the molecules in the double layer in terms of quantum mechanics by taking into account the possible form of the wave function of electron on the surface of the electrode.

### SECTION III

Title of Project:      The Study of the Dendritic Deposition of Zinc from Alkaline Solution

Long-term Techno-logical Aims:      To gain (a) a fuller understanding of the formation of zinc dendrites, (b) a mechanism which adequately describes the experimental behavior, and (c) the ability to apply simple equations to the growth of metal deposits in dendritic, or non-dendritic form as the system demands.

Specific Aims for this Period:      Submission of a paper concerning the capacitance study reported in a previous report.<sup>1</sup> Electron microscopic study to be performed. High purity system to be constructed, and high purity work to be initiated. Preliminary review of the work to be done in connection with the influence of surfactants upon dendritic growth.

New Evidence Revealed and Conclusions Reached:      An electron microscopic study by both transmission and scanning (by courtesy of I. B. M., East Fishkill Facility) techniques has revealed the following:  
  
(a) Zinc dendrite tip radii ( $r$ ) values are, for one overpotential, spread over a considerable range, e. g.,  $\eta = -85$  mv,  $r = 10^{-4}$  to  $10^{-5}$  cm, and

$\eta = -100$  mv,  $r = 4 \times 10^{-4}$  to  $10^{-4}$  cm. According to previous theory,<sup>2</sup> the values expected for  $r$  are  $5 \times 10^{-6}$  to  $10^{-4}$  cm at both overpotentials — the experimental is seen to be in accord with the theory.

(b) Scanning work has shown that the dendrite is essentially flat-sided; the cross-section could be best described as like that of a flat-bladed sword. The general dendrite tip area is both flat and stepped, except at the tip itself, which is of a spherical nature (as assumed in the theory<sup>2</sup>).

(c) Examination of spongy zinc up to 10,000 magnification appears to show that sponge is not micro-dendritic, as has been suggested.<sup>3</sup>

A high purity system was constructed based on zinc purification by amalgamation, and KOH purification by pre-electrolysis between gold electrodes. Preliminary experiments to ascertain KOH purity were promising, and it is expected that this work will be completed within the next report period.

Preliminary review into the field of surfactants has been concerned with those possible surfactants a basic study should include; the following have been chosen as among those to be investigated: Triton X-95, Triton X-100 and D. N. 65 (supplied to

us by Yardney Electric Corporation).

The paper concerned with the capacitance behavior during deposition was submitted to the Journal of Electroanalytical Chemistry; this paper has been returned with the referee's comments and is being amended in the light of those comments. The major paper on dendrite theory is expected to be submitted very shortly. Both papers are expected to be submitted before the next report period.

Specific Aims for  
the Next Report  
Period:

To ascertain whether dendrite growth in high purity systems differs from that reported previously<sup>2</sup> in impure systems. If differences are found, an experimental study similar to earlier work will be performed.

To initiate an experimental investigation into the influence of surfactants upon dendritic growth.

It is also proposed that, if the opportunity arises, a study on dendrite growth following dendrite pre-nucleation will be initiated — the object here will be to determine the validity of the theory concerning the total current-time phenomenon observed previously.<sup>2</sup>

### References

1. NASA Quarterly Repts. NsG-325. Periods of 1/1/68 - 3/31/68  
and 1/1/68 - 6/30/68.
2. NASA Rept. for the Period 7/1/67 - 12/21/67. NsG-325.  
Appendix A.
3. H. G. Oswin, private communication (Leesona Moos).

#### SECTION IV

Title of Project:	Reversibility of Organic Reactions
Long-term Fundamental Aim:	Investigation of the capability of organic compounds to be used in high energy secondary batteries.
Specific Aim for this Period:	The study of the relation between electrode kinetic parameters and the structure of organic compounds.
New Evidence Revealed and Con- clusions Reached:	Electrochemical reactions of organic compounds have been known for many years. With a few notable exceptions, practical applications of electro-organic reactions have not resulted. The application of hydrocarbon electro-oxidation in fuel cells, although very promising for high energy power sources, have not resulted (yet) in practical power sources due to the very low rate of reaction of the hydrocarbons. Some efforts to incorporate organic compounds in primary batteries have also been made with some prototype batteries produced with somewhat higher energy densities than existing primary cells. This study is directed toward the aim of increasing the reactivity of slow electro- organic couples for possible application in secondary batteries.

The series of compounds chosen as a model system is the para-substituted nitrosobenzenes and hydroxylamines. Their reactivity has been determined by the method of linear sweep voltammetry at platinum in 0.1 N  $\text{H}_2\text{SO}_4$  using a modification of Delahay's equations to determine  $E^0$  and  $i_0$ .

The compounds studied were commercially available and used without further purification. To obtain some of the compounds, the nitro derivative was reduced potentiostatically at the platinum electrode to obtain the hydroxylamine derivative. The substituents studied were: H, CHO,  $\text{CH}_2\text{CN}$ , CN,  $\text{COCH}_3$ ,  $\text{COCl}$ ,  $\text{COOH}$ ,  $\text{COC}_6\text{H}_5$ ,  $\text{OCH}_3$ ,  $\text{OCH}_2\text{COOH}$ ,  $\text{NH}_2$ ,  $\text{NO}_2$ ,  $\text{NCS}$ ,  $\text{N}(\text{CH}_3)_2$ ,  $\text{SO}_3\text{H}$ ,  $\text{SO}_2\text{Cl}$ , and  $\text{SO}_2\text{NH}_2$ . The experiments were all conducted in a conventional three-compartment electrochemical cell constructed from Pyrex and Teflon under an atmosphere of prepurified nitrogen. Potentials were measured against a saturated calomel electrode. Sweep rates varied from 4 to 1000 mV/sec.

The results show a considerable variation in  $E^0$  (0.25 V) and in  $i_0$  (one order of magnitude). Also the value of  $\partial V_{\text{peak}}/\partial \log \text{ sweep rate}$ , which is

equal to  $1/2$  the Tafel slope obtained in steady state measurements, varied with the substituent, indicating that different paths are followed or different steps are rate-determining. When they are divided into groups which apparently follow the same reaction path, meaningful correlations are possible.

The exchange current density increases exponentially with  $\sigma$ , the Hammett substituent constant, for all substituents studied irrespective of the reaction path. This observation is consistent with an interpretation that all paths involve a rate determining transition state which has a free radical characteristic. The more electron withdrawing substituents (positive  $\sigma$ ) interact with the free electron, stabilizing the radical and thus lowering the free energy of the activated complex. This would lead to an increased  $i_0$ , as is observed.

The reversible potential also varied linearly with  $\sigma$  but shows a strong dependence on the reaction mechanism with either a positive or negative slope observed. The positive slope corresponds to the reaction mechanism in which the nucleophilic attack is the rate determining step, while the



negative slope is due to an electrophilic attack as a rate determining step.

These results have shown that electro-organic couples can be modified, both in exchange current density and in their reversible potential by introducing suitable substituents into the molecule. The magnitude of the changes which may be produced is considerable (the practical range of  $\sigma$  values is four times the range utilized in these experiments), thus indicating a considerable potentiality for the production of high energy, high exchange current density electro-organic couples.

Specific Aims for  
the Next Report  
Period:

Steady state measurements of a few organic redox couples which in previous transient measurements proved to be reversible, in order to obtain additional kinetic data and evidence for the possible application to practical systems.

## SECTION V

Title of Project:      Ellipsometric Studies of Ion Adsorption on Solid  
Electrodes

Long-term Techno-      The purpose of this project is to study the adsorp-  
logical Aims:            tion of ions on electrodes of different metals by  
means of ellipsometry, and to investigate the  
the nature of the binding force between the ions  
and the electrode materials.

Specific Aims for      In this period prime efforts were made to prepare  
this Period:            optically uniform and stable electrode surfaces of  
different metals and to start some preliminary  
observation with these electrodes in the solutions  
of anions.

The metals currently under investigation are  
gold, silver, rhodium, and nickel. Ions under  
study include chloride, bromide, sulfate, and per-  
chlorate anions.

New Evidence            Electrodes of good optical quality could be obtained  
Revealed and Con-      from gold, silver, rhodium, and nickel by vacuum  
clusions Reached:        evaporation and sputtering onto glass slides.  
  
Efforts with lead and cadmium were, however,  
unsuccessful so far in forming mirrors of good

stability and optical quality.

Some preliminary ellipsometric measurements have been made with gold electrodes under potentiostatic conditions with a light of wavelength 5500 Å . Perchlorate ions show, at this wavelength, larger changes in the relative phase retardation with potential on gold electrode than on platinum electrode. Sulfate ions gave changes on gold electrode similar to those on platinum.

Primary Aims in  
the Next Period:

The surface coverages by the anions will be measured on the electrodes prepared. The possibility of using different wavelengths for different metal substrates will be tested. Attempts will be made to obtain lead and cadmium surfaces suitable for the ellipsometric measurements.

## SECTION VI

Title of Project:      Diffusion in Ceramics: Possibilities of High Diffusion Situation as Basis for Battery Involving Alkali Metal-Oxygen Cell

Long-term Technological Aims:      Development of high energy density storage battery.

Specific Aims for this Period:      Gathering of data on energy of activation of several sorts and systems; computer program will show how to put angles into atomic models so that model of certain ceramics can be built. Investigation of more literature concerning calculation method.

Conclusions Reached:      None.

Other:      New appointments have been made to this project which has been becalmed. Dr. Malcolm Fullenwider will contribute on a half-time basis. Mr. David LaPointe will put in 15-20 hours per week. Mr. LaPointe is a senior-year student whose time will not be charged to the project.

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